

## CERAMIC HEATER

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### Abstract

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**PROBLEM TO BE SOLVED:** To improve airtightness of a junction part and secure it for a long period of time by airtightly joining a coating plate of ceramic sintered product for covering the whole area of a junction surface of a substrate and the substrate of ceramic sintered product with an adhesive and forming an oxynitride glass layer containing specific elements on an junction interface.

**SOLUTION:** A substrate 2 of a ceramic sintered product provided with a recessed groove on its surface and a coating lower plate 3a of ceramic sintered product sandwich and support a heater element 8 in a recessed groove. Junction parts 7, 10 for airtightly joining between the substrate 2 and the coating lower plate 3a and between the coating lower plate 3a and an upper plate 3b for sandwiching and supporting the electrode plate 4 and the substrate 2 and the coating lower plate 3a are formed as an oxynitride glass layer, containing two kinds of elements selected from group IIIa of the periodic table, aluminum, and silicon so as to improve airtightness of the junction parts 7, 10 and secure airtightness over a long period of time. Thus, the glass layer of this composition has good wettability with the ceramic sintered product and has superior junction strength, satisfactory airtightness of the junction parts 7, 10, small dispersion in the junction strength, and superior heat resistance.

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[Title of the Invention]

## CERAMIC HEATER

[Abstract]

[Object] To provide a ceramic heater excellent in product life of which the reaction chamber is prevented from pollution by improving air-tightness in bonding parts and keeping the air-tightness for a long duration, of which the reaction chamber is prevented from pollution even when the air-tightness in the bonding parts is deteriorated, and of which the heater elements are not possibly disconnected.

[Solution] A ceramic heater comprises: a substrate made of a ceramic sintered body; a covering plate, made of a ceramic sintered body, for covering the entire region of a bonding face of the substrate; a heater element held between the substrate and the covering plate; and at least a pair of electrodes for feeding an electric power to a heater and having one end connected to the heater element, wherein the substrate and the covering plate are air-tightly bonded to each other by an adhesive, and an oxynitride glass layer containing at least two elements selected from group IIIa elements in the periodical table, aluminum and silicon is formed in the bonding interface.

[Claims]

1. A ceramic heater comprising: a substrate made of a ceramic sintered body; a covering plate, made of a ceramic sintered body, for covering

the entire region of a bonding face of the substrate; a heater element held between the substrate and the covering plate; and at least a pair of electrodes for feeding an electric power to a heater and having one end connected to the heater element, wherein the substrate and the covering plate are air-tightly bonded to each other by an adhesive, and an oxynitride glass layer containing at least two elements selected from group IIIa elements in the periodical table, aluminum and silicon is formed in the bonding interface.

2. The ceramic heater according to Claim 1, wherein the heater element is made of a silicon carbide sintered body, obtained by sintering without additive addition, having a sintering density of  $2.8 \text{ g/cm}^3$  or more and a specific electric resistance of  $0.1 \Omega\text{cm}$  or less at room temperature.

3. The ceramic heater according to Claim 1, wherein the ceramic sintered body is an aluminum nitride sintered body or an aluminum nitride-based sintered body.

4. The ceramic heater according to Claim 1, wherein an electrode plate, made of a metal, functioning as an electrode for plasma generation and/or electrode for electrostatic attraction by application of a high frequency voltage and/or direct current voltage is formed inside the covering plate.

#### [Detailed Description of the Invention]

##### [0001]

##### [Technical Field of the Invention]

The present invention relates to a ceramic heater which is installed in a reaction chamber, which is used for heating an object to be heated such

as a wafer without polluting inside the reaction chamber and excellent in durability.

[0002]

[Prior Art]

Conventionally, as a ceramic heater installed in a reaction chamber and used for heating an object to be treated such as a wafer or the like has been known a ceramic heater comprising a ceramic substrate having recessed grooves in the surface, heater elements made of a conductive ceramic packed in the recessed grooves, a covering plate made of a ceramic to mount the object thereon, and at least a pair of electrodes connected to the heater elements in one ends.

[0003]

[Problems]

With respect to such a conventional ceramic heater, the ceramic substrate and the covering plate made of a ceramic are bonded to each other by using various heat resistant adhesives. However, since they are burdened with high thermal load by repeated heating and cooling and exposed to high temperature atmosphere for a long duration, there occur the following problems.

[0004]

(1) Microcracks are easily formed in the bonding part and the air-tightness can not be maintained for a long duration. In a conventional heater element using a conductive ceramic, a sintering aid and an additive for giving conductivity (these additives are simply referred to as "additives") are added. However, these additives are evaporated and enter in a reaction

chamber through the bonding part whose air-tightness is lost to be pollutants of a semiconductor substrate.

[0005]

(2) In a conventional heater element using a conductive ceramic, since the additive is a different substance from the mother material, even dispersion of it is difficult; therefore, partial abnormal heat generation easily occurs and owing to the abnormal heat generation, a portion of the bonding layer is melted and leaking takes place in the bonding part to deteriorate the air-tightness. Further, similarly to the above-mentioned problem, the additive becomes a pollutant of a semiconductor substrate.

[0006]

(3) In a conventional heater element using a conductive ceramic, since the high temperature strength is insufficient, deformation and damages are easily caused by thermal impact.

[0007]

[Problems to be Solved by the Invention]

The present invention is accomplished in consideration of the above-mentioned problems of conventional techniques, and an object of the present invention is to provide a ceramic heater with a long product life and with which a risk of pollution in the inside of a reaction chamber is eliminated by improving the air-tightness in a bonding part and, at the same time, the air-tightness is kept for a long duration; pollution in the inside of the reaction chamber is avoided even when the air-tightness in the bonding part is deteriorated; and a risk of disconnection of heat elements is eliminated.

[0008]

[Means for Solving the Problems]

As practical means for effectively solving the above-mentioned problems, a ceramic heater according to Claim 1 is a ceramic heater comprising: a substrate made of a ceramic sintered body; a covering plate, made of a ceramic sintered body, for covering the entire region of a bonding face of the substrate; a heater element held between the substrate and the covering plate; and at least a pair of electrodes for feeding an electric power to a heater and having one end connected to the heater element, wherein the substrate and the covering plate are air-tightly bonded to each other by an adhesive, and an oxynitride glass layer containing at least two elements selected from group IIIa elements in the periodical table, aluminum and silicon is formed in the bonding interface.

[0009]

In a ceramic heater according to Claim 2, the heater element is made of a silicon carbide sintered body, obtained by sintering without additive addition, having a sintering density of 2.8 g/cm<sup>3</sup> or more and a specific electric resistance of 0.1 Ωcm or less at room temperature.

[0010]

In a ceramic heater according to Claim 3, the ceramic sintered body is an aluminum nitride sintered body or an aluminum nitride-based sintered body.

[0011]

In a ceramic heater according to Claim 4, an electrode plate, made of a metal, functioning as an electrode for plasma generation and/or electrode

for electrostatic attraction by application of a high frequency voltage and/or direct current voltage is formed inside the covering plate.

[0012]

**[Embodiments of the Invention]**

Hereinafter, embodiments of the present invention will be described with reference to the drawings. The descriptions of the embodiments are only for enabling the present invention to be understood purely by way of non-limiting example and it is not intended that the present invention be limited to the illustrated embodiments.

[0013]

In this embodiment, as illustrated in Fig. 1 and Fig. 2, a ceramic heater 1 comprises a substrate 2 made of a ceramic sintered body and having recessed grooves 12 in the surface, heater elements 8 packed in the recessed grooves, a covering plate 3 made of a ceramic sintered body covering the entire region of the substrate 2 and the heater elements 8 and mounting an object particle to be treated thereon, electrodes 9 made of nickel excellent in heat resistance and connected to the heater elements in one ends for feeding an electric power to the heater elements, and a thermocouple 6 for measuring the temperature of the covering plate 3 for mounting an object to be heated thereon.

[0014]

The substrate 2 and the covering plate 3 are air-tightly bonded to each other by a glassy adhesive and an oxynitride glass layer containing at least two elements selected from group IIIa elements, aluminum and silicon is formed in the bonding interface and the thickness of the bonding layer 10

after bonding is controlled to be 5 to 180  $\mu\text{m}$ .

[0015]

Further, the heater elements 8 are made of a silicon carbide sintered body obtained by sintering without additive addition and having a sintered density of  $2.8 \text{ g/cm}^3$  and a specific electric resistance of  $0.1 \Omega\text{cm}$  or less at room temperature.

[0016]

Further, the ceramic sintered body is formed from an aluminum nitride sintered body or an aluminum nitride-based sintered body since they are excellent in the thermal conductivity, mechanical strength and durability to plasma, and the aluminum nitride sintered body or the aluminum nitride-based sintered body to be used is produced by a conventional method.

[0017]

Further, the covering plate 3 comprises a covering lower plate 3a made of a ceramic sintered body having recessed grooves 11 in the top face, an electrode plate 4 made of a metal and filling the recessed grooves 11, a covering upper plate 3b made of a ceramic sintered body and covering the entire region of the covering lower plate 3a and the electrode plate 4, and electrode 5 of nickel excellent in heat resistance and connected to the electrode plate 4 in one end for applying high frequency voltage and/or direct current voltage. High frequency voltage for plasma generation and/or direct current voltage for electrostatic attraction is applied to the electrode plate 4 so as to make the electrode plate 4 functioning as an electrode for plasma generation and/or an electrode for electrostatic

attraction.

[0018]

The covering lower plate 3a and the covering upper plate 3b are air-tightly bonded by a glassy adhesive and an oxynitride glass layer containing at least 2 elements selected from group IIIa elements in the periodical table, aluminum and silicon is formed in their bonding interface. The thickness of the bonding layer 7 after bonding is controlled to be 5 to 180  $\mu$ m.

[0019]

Hereinafter, the requirements for the respective components composing the present invention will be described in detail.

[Glassy adhesive]

The following is the reason for forming the oxynitride glass layers containing at least two elements selected from group IIIa elements in the periodical table, aluminum and silicon as glassy adhesive layers for air-tightly bonding the substrate 2 and the covering lower plate 3a holding the heater elements 8 and the covering lower plate 3a and the covering upper plate 3b holding the electrode plate 4. Owing to the formation of the glassy bonding layers, the air-tightness of the bonding parts 7 and 10 can be remarkably improved and the air-tightness can be kept for a long duration.

[0020]

That is, the oxynitride glass having the above-mentioned components has good wettability to a ceramic sintered body, excellent bonding property, good air-tightness in the bonding parts 7 and 10, narrow dispersion of the bonding strength, and excellent heat resistance.

[0021]

The thermal expansion coefficient of the oxynitride glass containing the above-mentioned components is  $3 \times 10^{-6}$  to  $8 \times 10^{-6}/^{\circ}\text{C}$  and almost same as or close to the thermal expansion coefficient ( $3.8 \times 10^{-6}$  to  $4.7 \times 10^{-6}/^{\circ}\text{C}$ ) of an aluminum nitride sintered body which is preferably used among ceramic sintered bodies. Accordingly, damages of the bonding layers 7 and 10 by repeated temperature increase and decrease and thermal stress at the time of application of heat load of cooling, that is, occurrence of cracking, can be avoided. Therefore, the air-tightness of the bonding layers 7 and 10 can be maintained for a long duration.

[0022]

The glass softening point  $T_g$  of the oxynitride glass layer containing the above-mentioned components is as high as 850 to 950°C and the bonding layers 7 and 10 are not deteriorated even after exposure to high temperature atmosphere for a long duration.

[0023]

The adhesives capable of forming the oxynitride glass layers can be produced as follows, for example. At first, silicon dioxide and aluminum oxide or compounds to be these oxides by heating are mixed with oxides of at least two elements selected from group IIIa elements in the periodical table as raw material powders for a bonding material. The oxides of group IIIa elements in the periodical table are not particularly limited and may include  $\text{Y}_2\text{O}_3$ ,  $\text{Dy}_2\text{O}_3$ ,  $\text{Er}_2\text{O}_3$ ,  $\text{Gd}_2\text{O}_3$ ,  $\text{La}_2\text{O}_3$ ,  $\text{Yb}_2\text{O}_3$ ,  $\text{Sm}_2\text{O}_3$  and the like.

[0024]

Among them, in view of the cost and the availability,  $\text{Y}_2\text{O}_3$  is most

preferable as one of the oxides of group IIIa elements in the periodical table and  $\text{Dy}_2\text{O}_3$ ,  $\text{Er}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$  easy to form full solid-solution with  $\text{Y}_2\text{O}_3$  are preferable as other oxides of group IIIa elements in the periodical table and  $\text{Dy}_2\text{O}_3$  is particularly preferable in view of the cost.

[0025]

The composition ratios of the above-mentioned respective components are not particularly limited, however it is preferable to mix these components so as to obtain a melt containing two elements selected from group IIIa elements in the periodical table in the total amount of 20 to 50% by weight in terms of oxide, silicon dioxide in an amount of 30 to 70% by weight, and aluminum oxide 10 to 30% by weight since the obtained melt has a low melting point and excellent wettability to a ceramic member or the like. Further, in the case the elements selected from group IIIa elements in the periodical table are two elements, it is preferable to mix oxides of these two group IIIa elements at about 1 : 1 mole ratio since the melting temperature of the obtained bonding material becomes lowest.

[0026]

The above-mentioned raw material mixed powder is pulverized to, for example, 5  $\mu\text{m}$  or less and, after being melted at 1500 to 1700°C, the raw material mixture was quenched to obtain a glassy cooled material and the material is again pulverized to about 5  $\mu\text{m}$ <sup>10-14</sup> to obtain an adhesive of the molten fine powder with a uniform composition. The atmosphere for the production of the adhesive is not particularly limited. However, when the production steps are carried out in nitrogen atmosphere, oxynitride glass is formed and if in non-nitrogen atmosphere, oxide glass is formed.

[0027]

However, in the present invention, it is indispensable to form an oxynitride glass layer in the bonding interface, so that it is preferable to carry out the steps of producing the bonding material in nitrogen-containing atmosphere so as to previously vitrify the bonding material to be oxynitride glass. The nitrogen-containing atmosphere may be produced using N<sub>2</sub> gas, H<sub>2</sub>-N<sub>2</sub> mixed gas or NH<sub>3</sub> gas.

[0028]

Further, Si<sub>3</sub>N<sub>4</sub> powder and/or AlN powder in an amount of 1 to 50% by weight out of the total weight is preferable to be added to the above-mentioned bonding material. That is, addition of Si<sub>3</sub>N<sub>4</sub> powder and/or AlN powder decreases the thermal expansion coefficient of the oxynitride glass and, also, improves the heat resistance. When the addition amount is less than 1% by weight, the addition is meaningless and when it exceeds 50% by weight, the adhesion strength is inversely decreased. The particle size of the Si<sub>3</sub>N<sub>4</sub> powder and/or AlN powder to be added is not particularly limited; however, the powder with an average particle diameter of 0.8 μm or less is preferable in view of formation of oxynitride glass with an even concentration.

[0029]

[Thickness of bonding layer]

The reason for adjusting the thickness of the bonding layer 10 of the substrate 2 and the covering plate 3 after bonding to be preferably 5 to 180 μm and the thickness of the bonding layer 7 of the covering lower plate 3a and the covering upper plate 3b after bonding to be preferably 5 to 180 μm

is as follows and the air-tightness of the bonding parts can be firmly maintained.

[0030]

That is, when the thickness of the bonding layer 10 is less than 5  $\mu\text{m}$ , fillet formation in the end parts of the bonding layer 10 is insufficient. Therefore, air-tightness of the bonding part 10 cannot be secured and the bonding strength is also insufficient. On the other hand, when the thickness of the bonding layer 10 exceeds 180  $\mu\text{m}$ , although the air-tightness of the bonding layer 10 can be secured, the bonding strength tends to be easily decreased. Owing to the heating at the time of bonding, the melted glassy adhesive flows out of the end parts of the bonding layer 10 to make it impossible to bond the substrate 2 and the covering lower plate 3a in parallel. Accordingly, the production yield is decreased and the bonding work is possibly interrupted in some cases.

[0031]

Meanwhile, when the thickness of the bonding layer 7 is less than 5  $\mu\text{m}$ , fillet formation in the end parts of the bonding layer 7 is insufficient. Therefore, air-tightness of the bonding part 7 cannot be secured and the bonding strength is also insufficient. On the other hand, when the thickness of the bonding layer 7 exceeds 180  $\mu\text{m}$ , although the air-tightness of the bonding layer 7 can be secured, the bonding strength tends to be easily decreased. Owing to the heating at the time of bonding, the melted glassy adhesive flows out of the end parts of the bonding layer 7 to make it impossible to bond the covering lower plate 3a and the covering upper plate 3b in parallel. Accordingly, the production yield is decreased and the

bonding work is possibly interrupted in some cases.

[0032]

[Heater element]

The material for the above-mentioned heater elements 8 preferably comprises a silicon carbide sintered body obtained by sintering without additive addition and having a sintered density of 2.8 g/cm<sup>3</sup> and a specific electric resistance of 0.1 Ωcm or less at room temperature, because of the following reasons. The covering plate 3 can be maintained at a prescribed temperature by applying electric power to the heater elements 8.

[0033]

The heater elements 8 made of such a silicon carbide sintered body can be produced by any of the following summarized methods described, for example, in JP Kokai Hei 4-65361.

(1) A first silicon carbide powder with an average particle diameter of 0.1 to 10 μm and a second silicon carbide powder having an average particle diameter of 0.1 μm or less and obtained by introducing raw material gases consisting of a silane compound or a halogenated silicon together with a hydrocarbon into plasma in non-oxidizing atmosphere and carrying out vapor phase deposition while controlling the pressure of the reaction system to be less than one atmospheric pressure to 0.1 torr are mixed and sintered by firing to obtain a silicon carbide sintered body and the sintered body is subjected to electric discharge processing according to desired patterns to obtain heater elements.

[0034]

(2) A silicon carbide powder having an average particle diameter of

0.1  $\mu\text{m}$  or less and obtained by introducing raw material gases consisting of a silane compound or a halogenated silicon together with a hydrocarbon into plasma in non-oxidizing atmosphere and carrying out vapor phase deposition while controlling the pressure of the reaction system to be less than one atmospheric pressure to 0.1 torr is sintered by firing to obtain a silicon carbide sintered body and the sintered body is subjected to electric discharge processing according to desired patterns to obtain heater elements.

[0035]

Accordingly, since these heater elements 8 are made of silicon carbide sintered bodies obtained by sintering without additive addition, that is, without foreign substance addition, they are even. Consequently, no partial abnormal heat generation takes place and partial melting of the bonding layers 7 and 10 and consequent leakage in the bonding parts are not caused. As a result, the air-tightness of the bonding layers 7 and 10 can be further reliably secured.

[0036]

Since the sintered bodies are extremely pure owing to no additive addition and have a density as high as 2.8 g/cm<sup>3</sup> or more sintering density, even when leakage occurs in the bonding parts and the air-tightness is deteriorated, no additive, that is no impurity, is evaporated from the heater elements 8 and for that, there is no risk of pollution in a reaction chamber. Further, since the sintered bodies are excellent also in the high temperature strength, deformation and disconnection of the heater elements 8 by thermal impacts do not occur and also since the specific electric resistance is

as low as 1  $\Omega$ cm or less at a room temperature, it is not required for the heater elements 8 to be made fine and thin and therefore, there is no risk of disconnection of the heater elements 8.

[0037]

[Electrode plate]

Plasma can be generated by applying high frequency voltage to the electrode plate 4 through an electrode 5 for application from a plasma generation power source. At that time, since the electrode plate 4 is a metal plate having a sufficient thickness  $T_1$  of 0.025 mm or more, it has the following advantageous points: (1) there is no risk of burning out and disconnection by heat generation even when high frequency voltage is applied; (2) plasma can be densely, stably and evenly generated in the whole region unlike the case of plasma generation by using a lattice type or mesh type electrode; and (3) connection of the electrode plate 4 and the electrode 5 for application can be reliably formed between a plane and a rod.

[0038]

When a direct current high voltage of about 500 V is applied to the foregoing electrode plate 4 through the electrode 5 for application from an electrostatic attraction power source, the covering plate 3 functions as an insulator and an object to be attracted such as a silicon wafer can be electrostatically attracted. In the case high frequency voltage from a power source for generating plasma and direct current high voltage from a power source for electrostatic attraction are applied to the electrode plate 4, a filter capable of cutting the high frequency may be installed between the power source for electrostatic attraction and the electrode for power application.

[0039]

The above-mentioned electrode plate 4 is preferably made of a high melting point metal such as molybdenum, tungsten, tantalum, niobium or an alloy thereof because (1) the plate is provided with a thermal expansion coefficient close to that of a ceramic; (2) it is stable at a heating temperature and in the atmosphere in the bonding step; (3) it has a low specific resistance; and (4) it can be used for a long duration in a practical use temperature range from room temperature to 1000°C.

[0040]

[Ceramic sintered body]

Materials for the above-mentioned substrate 2, covering lower plate 3a, and covering upper plate 3b are not particularly limited; however, they are preferably formed from an aluminum nitride sintered body or an aluminum nitride-based sintered body owing to high thermal conductivity, mechanical strength, resistance to plasma, and durability to plasma cleaning gases such as  $\text{CF}_4$ ,  $\text{C}_2\text{F}_6$ ,  $\text{C}_3\text{F}_8$ . Those produced by known methods can be used as the aluminum nitride sintered body and the aluminum nitride-based sintered body.

[0041]

The ceramic heater according to this embodiment can be produced as follows, for example. The substrate 2 and the covering lower plate 3a having recessed grooves 12 in the surface are formed by pressing a die having projected parts in the surface to ceramic formed bodies and sintering the formed bodies. The sintering conditions such as sintering temperature may be controlled according to those in a conventional method. The

covering upper plate 3 is also formed by a conventional method. The electrode plate 4 and the heater elements 8 formed by the above-mentioned method are arranged so as to fill the recessed grooves 11 of the covering lower plate 3a and the recessed grooves 12 of the substrate 2, respectively.

[0042]

Next, the above-mentioned finely pulverized glassy adhesive is mixed with a screen oil to produce a paste and the paste type glassy adhesive is applied to the respective bonding faces of the substrate 2 whose recessed grooves 12 are filled with heater elements 8 and the covering lower plate 3a as well as to the respective bonding faces of the covering lower plate 3a whose recessed grooves 11 are filled with the electrode plate 4 and the covering upper plate 3b and dried at 100 to 200°C.

[0043]

After that, while holding the heater elements 8 and the electrode plate 4, through the faces coated with the glassy adhesive, the substrate 2, the covering lower plate 3a, and the covering upper plate 3b are laminated and the glassy adhesive is melted by heating in an electric furnace and bonding is performed by heating at 1300 to 1500°C for 5 to 40 minutes.

[0044]

The heating is carried out in normal pressure or under pressure of about 10 atmospheric pressure or less. The atmosphere at the time of heating differs depending on the adhesive to be employed. That is, in the case of using an adhesive containing oxynitride glass, since the adhesive is previously converted into oxynitride, the atmosphere may be no nitrogen-containing atmosphere. However, nitrogen-containing

atmosphere is preferable. On the other hand, in the case where an oxide glass-containing adhesive is used, a nitrogen source is required for converting the oxide glass into oxynitride and heating is carried out in nitrogen-containing atmosphere. The nitrogen-containing atmosphere can be produced by using N<sub>2</sub> gas, H<sub>2</sub>-N<sub>2</sub> mixed gas or NH<sub>3</sub> gas.

[0045]

The heated and melted adhesive is provided with bonding strength by cooling and solidifying treatment and it is preferable to gradually cool the adhesive but not quench it quickly so as to keep the intake of nitrogen high and stabilize the oxynitride glass layers. The cooling rate is preferably 50°C/min or less, more preferably 30°C/min or less.

[0046]

In order to adjust the thickness of the bonding layers 7 and 10 after bonding to be in the above-mentioned range, the treatment conditions such as the amount ratios of the finely pulverized glassy adhesive and the screen oil, the application amount of the paste type glassy adhesive, the heating temperature at the time of bonding, the duration are properly controlled.

[0047]

[Examples]

Hereinafter, examples of the present invention will be described in detail.

[Examples 1 to 5]

A substrate made of an aluminum nitride sintered body having a diameter of 220 mm, a thickness of 15 mm and spiral recessed grooves with a width of 5 mm and a depth of 3 mm in the surface for disposing heater

elements therein; a covering lower plate made of an aluminum nitride sintered body having a diameter of 220 mm, a thickness of 8 mm and a circular recessed groove with a diameter of 200 mm and a depth of 3 mm in the surface for disposing an electrode plate therein; and a covering upper plate made of an aluminum nitride sintered body with a diameter of 220 mm and a thickness of 1 mm were produced by a conventional method.

[0048]

On the other hand, heater elements with a shape to be disposed in the above-mentioned spiral recessed grooves and made of a silicon carbide sintered body obtained by sintering without addition of a sintering aid or additive for providing conductivity and having a sintered density of 3.1 g/cm<sup>3</sup> and a specific electric resistance of 0.05 Ωcm at room temperature were produced by the production method (1) described in the paragraph [0031]. The average particle diameter of the first silicon carbide powder was 0.7 μm and the addition amount was 95% by weight; the average particle diameter of the second silicon carbide was 0.01 μm and the addition amount was 5% by weight; and the hot press sintering conditions were 400 kg/cm<sup>2</sup> press pressure, 2200°C sintering temperature, and 90 minute sintering duration. As the electrode plate, a molybdenum metal plate with a diameter of 200 mm and a thickness of 0.3 mm was made ready.

[0049]

Next, glassy adhesives (particle diameter of about 2 μm) having the compositions shown in Table 1 were mixed with a commercialized screen oil to produce pastes and the obtained paste type glassy adhesives were applied to the respective bonding faces of the substrate of the ceramic sintered body

whose recessed grooves were filled with heater elements and the above-mentioned covering lower plate of the ceramic sintered body as well as to the respective bonding faces of the above-mentioned covering lower plate of the ceramic sintered body whose recessed groove was filled with the electrode plate of molybdenum and the above-mentioned covering upper plate of the ceramic sintered body and dried at 100 to 200°C.

[0050]

After that, while holding the above-mentioned heater elements and electrode plate, through the faces coated with the glassy adhesive, the above-mentioned substrate lower plate made of the ceramic sintered body, the above-mentioned substrate upper plate made of the ceramic sintered body, and the above-mentioned covering plate made of the ceramic sintered body were laminated and the glassy adhesive was melted by heating in an electric furnace and air-tight bonding was performed by heating at 1400°C for 20 minutes. The cooling rate was 25°C/min and the thickness of both bonding layers after the bonding was both 50 µm. Formation of oxynitride glass in the bonding interfaces was confirmed by Auger electron spectroscopy.

[0051]

In order to confirm the air-tightness of the bonding part of the substrate made of the aluminum nitride sintered body and the covering lower plate made of the aluminum nitride sintered body in the ceramic heater obtained in such a manner, each obtained heater was subjected to a durability test. The results of the test are shown in Table 1. Summary of the durability test was as follows.

[0052]

Each ceramic heater was heated from a room temperature to the highest temperature, 700°C, in 1 hour by applying electricity to the ceramic heater and kept at the highest temperature for 30 minutes and then gradually cooled to room temperature. The air-tightness of the foregoing bonding part was tested by a leak test using He gas after such a heat cycle was repeated 100 times. The evaluation standards of the air-tightness were as follows.

○: the leakage amount of He was  $10^{-9}$  torr.l/sec or less:

△: the leakage amount of He was  $10^{-9}$  to  $10^{-8}$  torr.l/sec: and

×: the leakage amount of He was  $10^{-8}$  torr.l/sec or more.

[0053]

[Table 1]

	Adhesive raw material powder composition (% by weight)				Air-tightness
	Dy <sub>2</sub> O <sub>3</sub>	Y <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	
Example 1	15	10	25	50	○
Example 2	15	15	25	45	○
Example 3	15	15	20	50	○
Example 4	20	20	20	40	○
Example 5	25	25	15	35	○

[0054]

[Comparative Examples 1 to 4]

Ceramic heaters were produced according to Examples 1 to 5 and

subjected to the durability test. The glassy adhesive compositions employed were as shown in Table 2.

[0055]

[Table 2]

	Adhesive raw material powder composition (% by weight)				Air-tightness
	Dy <sub>2</sub> O <sub>3</sub>	Y <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	
Comparative Example 1	0	20	40	40	×
Comparative Example 2	0	35	20	45	△
Comparative Example 3	0	40	10	50	×
Comparative Example 4	20	0	40	40	×
Comparative Example 5	35	0	20	45	△
Comparative Example 6	40	0	10	50	×

[0056]

[Effects of the Invention]

As described above, a ceramic heater according to the present invention comprises: a substrate made of a ceramic sintered body; a covering plate, made of a ceramic sintered body, for covering the entire region of a bonding face of the substrate; a heater element held between the substrate and the covering plate; and at least a pair of electrodes for feeding an electric power to a heater and having one end connected to the heater

element, wherein the substrate and the covering plate are air-tightly bonded to each other by an adhesive, and an oxynitride glass layer containing at least two elements selected from group IIIa elements in the periodical table, aluminum and silicon is formed in the bonding interface. Therefore, the air-tightness in the bonding part is remarkably improved; the air-tightness is kept for a long duration; pollution in the inside of the reaction chamber can be avoided; no pollution is caused in the inside of a reaction chamber even if the air-tightness in the bonding parts is deteriorated; disconnection of the heater elements is prevented; and product life can be prolonged.

[0057]

In a ceramic heater according to Claim 2, the heater element is made of a silicon carbide sintered body, obtained by sintering without additive addition, having a sintering density of 2.8 g/cm<sup>3</sup> or more and a specific electric resistance of 0.1 Ωcm or less at room temperature. Therefore, even when the air-tightness in the bonding parts is deteriorated, no pollution attributed to evaporation of additives is caused in the inside of the reaction chamber and since heater elements are excellent in the high temperature strength, their disconnection can be prevented to result in a long life.

[0058]

In a ceramic heater according to Claim 3, the ceramic sintered body is an aluminum nitride sintered body or an aluminum nitride-based sintered body. Therefore, the ceramic heater is excellent in the thermal conductivity, mechanical strength and resistance to plasma, and is provided with improved durability and a long product life.

[0059]

In a ceramic heater according to Claim 4, an electrode plate, made of a metal, functioning as an electrode for plasma generation and/or electrode for electrostatic attraction by application of a high frequency voltage and/or direct current voltage is formed inside the covering plate. Therefore, plasma can be generated by applying high frequency voltage and the covering plate functions as an insulator by applying a direct current voltage to make electrostatic attraction of an object to be treated such as a silicon wafer possible.

[Brief Description of the Drawings]

Fig. 1 is a vertical cross-sectional view of a ceramic heater according to an embodiment of the present invention.

Fig. 2 is a vertical cross-sectional exploded view for illustrating a ceramic heater according to another embodiment of the present invention.

[Explanation of Symbols]

- 1 ceramic heater
- 2 substrate
- 3 covering plate made of ceramic sintered body
- 3a covering lower plate
- 3b covering upper plate
- 4 electrode plate made of metal
- 5 electrode for applying high frequency and/or direct current voltage
- 6 thermocouple
- 7 bonding layer
- 8 heater element

9 electrode for feeding electric power to heater

10 bonding layer

11, 12 recessed grooves

Fig.1

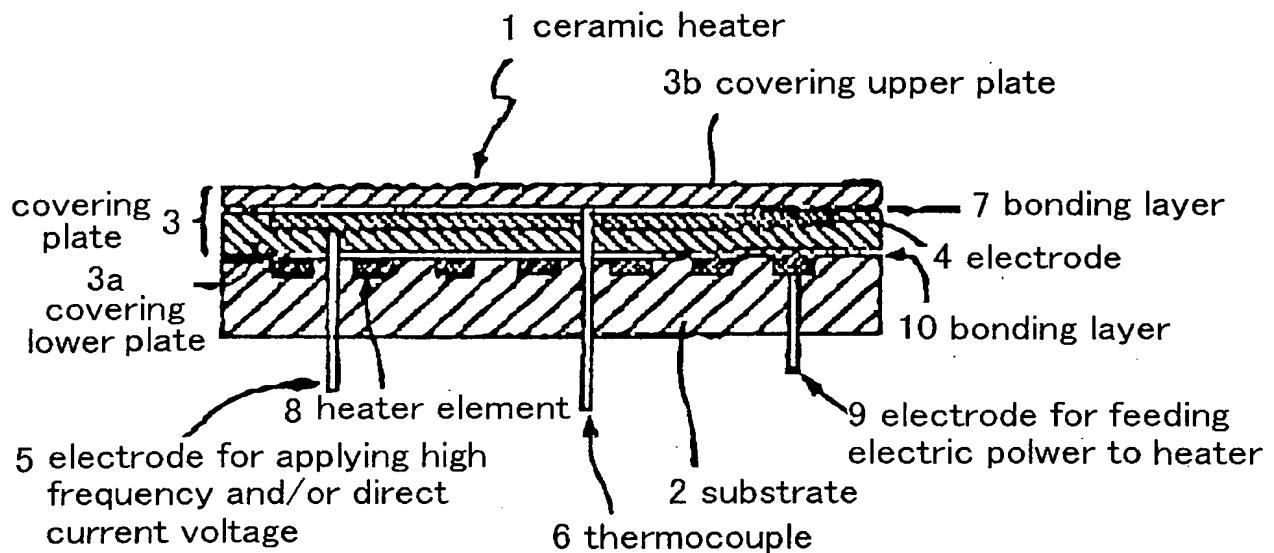
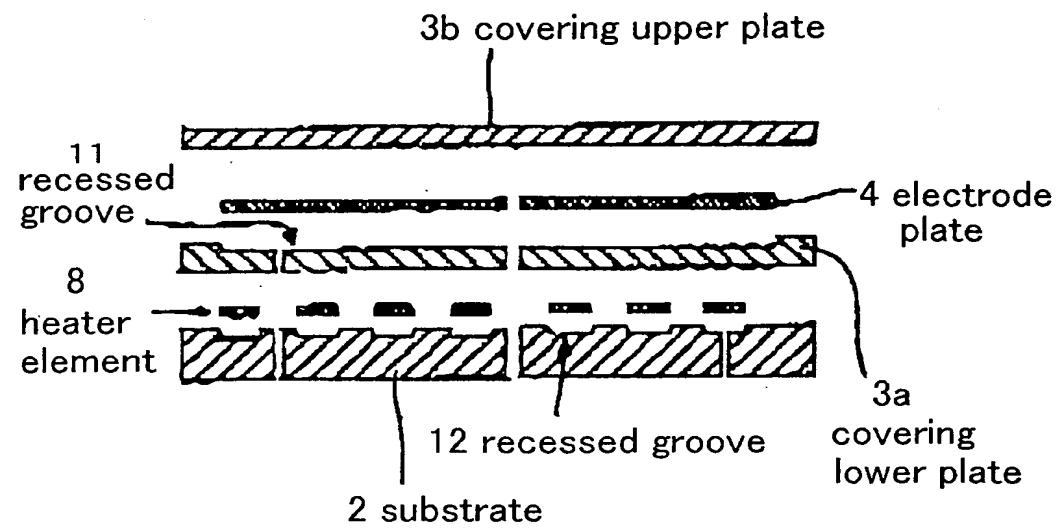


Fig.2



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(54)【発明の名称】 セラミックスヒータ

## (57)【要約】

【課題】 接合部の気密性を向上させると共に、その気密性を長期に亘って確保することにより、反応チャンバー内が汚染される虞がなく、また接合部の気密性が損なわっても反応チャンバー内が汚染される虞がなく、更にヒータエレメントが破断する虞がない、製品寿命に優れたセラミックスヒータを提供することを課題とする。

【解決手段】 セラミックス焼結体製の基体と、この基体の接合面の全領域を覆うセラミックス焼結体製の被覆板とを備えるとともに、前記基体と前記被覆板との間に把持されたヒータエレメントと、このヒータエレメントに一端が接続された少なくとも1対のヒータ給電用電極とを備え、前記基体と前記被覆板とは接合剤により気密に接合され、その接合界面には周期表第 IIIa 属元素から選ばれた少なくとも2種の元素と、アルミニウムと、珪素とを含むオキシナイトライドガラス層が形成されているように構成する。

## 【特許請求の範囲】

【請求項1】セラミックス焼結体製の基体と、この基体の接合面の全領域を覆うセラミックス焼結体製の被覆板とを備えるとともに、前記基体と前記被覆板との間に把持されたヒータエレメントと、このヒータエレメントの一端が接続された少なくとも1対のヒータ給電用電極とを備え、前記基体と前記被覆板とは接合剤により気密に接合され、その接合界面には周期表第 IIIa 属元素から選ばれた少なくとも2種の元素と、アルミニウムと、珪素とを含むオキシナイトライドガラス層が形成されていることを特徴とするセラミックスヒータ。

【請求項2】前記ヒータエレメントは、添加剤が添加されることなく焼結され、焼結密度が $2.8\text{ g/cm}^3$ 以上、室温での電気比抵抗が $0.1\Omega\text{ cm}$ 以下の炭化珪素焼結体からなることを特徴とする請求項1に記載のセラミックスヒータ。

【請求項3】前記セラミックス焼結体は窒化アルミニウム焼結体または窒化アルミニウム基焼結体であることを特徴とする請求項1に記載のセラミックスヒータ。

【請求項4】前記被覆板の内部に、高周波電圧及び/又は直流電圧の印加によりプラズマ発生用電極及び/又は静電吸着用電極として作用する金属製の電極板を設けたことを特徴とする請求項1に記載のセラミックスヒータ。

## 【発明の詳細な説明】

## 【0001】

【発明が属する技術分野】本発明は、反応チャンバー内に設置され、ウェハ等の被加熱物の加熱に用いられて、反応チャンバー内を汚染させることがなく、耐久性にも優れたセラミックスヒータに関する。

## 【0002】

【従来の技術】従来、反応チャンバー内に設置され、ウェハ等の被加熱物の加熱に用いられるセラミックスヒータとしては、表面に凹溝を設けられたセラミックス製基板と、該凹溝に充填された導電性セラミックス製のヒータエレメントと、前記基板と前記ヒータエレメントの全領域を覆い、被加熱物を載置するセラミックス製被覆板と、前記ヒータエレメントに一端が接続された少なくとも1対の電極を少なくとも備えたものが知られている。

【0003】【問題点】この従来のセラミックスヒータにあっては、前記セラミックス製基板と前記セラミックス製被覆板とは各種の耐熱性接合剤を用いて接合されているが、繰り返しの昇温、降温の熱負荷や、高温雰囲気に長時間晒されるため、次のような問題点が惹起していた。

【0004】① 接合部に微細なクラックが入りやすく、気密性を長期に亘り確保できない。導電性セラミックスを用いた従来のヒータエレメントにあっては、焼結助剤や導電性を付与するための添加剤（これらの添加剤を単に「添加剤」という）が添加されているが、これら

の添加剤が蒸発し、気密性が失われた前記接合部を通じて反応チャンバー内に流入し、半導体基板の汚染源となる。

【0005】② 導電性セラミックスを用いた従来のヒータエレメントは、添加剤は母材とは異種物質であるため均一分散化が困難であり、そのため局所的な異常発熱が起こりやすく、この異常発熱により接合層の一部が溶融して前記接合部にリーキが生じて気密性が損なわれ、前記と同様に、前記添加剤が半導体基板の汚染源となる。

【0006】③ 導電性セラミックスを用いた従来のヒータエレメントは、高温強度が充分でないため、熱衝撃による変形、破損が起こりやすい。

## 【0007】

【発明が解決しようとする課題】本発明は、従来技術の前記問題点に鑑みてなされたものであり、その課題とするところは、接合部の気密性を向上させると共に、その気密性を長期に亘って確保することにより、反応チャンバー内が汚染される虞がなく、また接合部の気密性が損なわっても反応チャンバー内が汚染される虞がなく、更にヒータエレメントが破断する虞がない、製品寿命に優れたセラミックスヒータを提供することにある。

## 【0008】

【課題を解決するための手段】前記課題を効果的に解決できる具体的に構成した手段として、請求項1に係るセラミックスヒータは、セラミックス焼結体製の基体と、この基体の接合面の全領域を覆うセラミックス焼結体製の被覆板とを備えるとともに、前記基体と前記被覆板との間に把持されたヒータエレメントと、このヒータエレメントの一端が接続された少なくとも1対のヒータ給電用電極とを備え、前記基体と前記被覆板とは接合剤により気密に接合され、その接合界面には周期表第 IIIa 属元素から選ばれた少なくとも2種の元素と、アルミニウムと、珪素とを含むオキシナイトライドガラス層が形成されていることを特徴とするものである。

【0009】また、請求項2に係るセラミックスヒータは、前記ヒータエレメントは、添加剤が添加されることなく焼結され、焼結密度が $2.8\text{ g/cm}^3$ 以上、室温での電気比抵抗が $0.1\Omega\text{ cm}$ 以下の炭化珪素焼結体からなることを特徴とする。

【0010】また、請求項3に係るセラミックスヒータは、前記セラミックス焼結体は窒化アルミニウム焼結体または窒化アルミニウム基焼結体であることを特徴とする。

【0011】また、請求項4に係るセラミックスヒータは、前記被覆板の内部に、高周波電圧及び/又は直流電圧の印加によりプラズマ発生用電極及び/又は静電吸着用電極として作用する金属製の電極板を設けたことを特徴とする。

## 【0012】

【発明の実施の形態】以下、本発明の実施の形態について図示説明する。ただし、この実施の形態は、発明の趣旨をより良く理解させるため具体的に説明するものであり、特に指定のない限り、発明内容を限定するものではない。

【0013】この実施の形態において、図1および図2に示すように、セラミックスヒータ1は、表面に凹溝12が設けられたセラミックス焼結体製の基体2と、凹溝に装填されたヒータエレメント8と、基体2とヒータエレメント8の全領域を覆い、被加熱物を載置するセラミックス焼結体製の被覆板3と、ヒータエレメント8に一端が接続され、耐熱性に優れたニッケルにより形成されたヒータ給電用電極9と、被加熱物を載置する被覆板3の温度を測定する熱電対6とを備えている。

【0014】そして、基体2と被覆板3とはガラス質接合剤により気密に接合され、その接合界面には周期表第IIIa族元素から選ばれた少なくとも2種の元素と、アルミニウムと、珪素を含むオキシナイトライドガラス層が形成されており、接合後の接合層10の厚みが5~180μmとなるよう構成されている。

【0015】また、ヒータエレメント8は、添加剤が添加されることなく焼結され、焼結密度が2.8g/cm<sup>3</sup>、室温での電気比抵抗が0.1Ωcm以下の炭化珪素焼結体から形成されている。

【0016】更に、前記セラミックス焼結体は、熱伝導性、機械的強度、耐プラズマ性に優れることから窒化アルミニウム焼結体、または窒化アルミニウム基焼結体により形成されており、この窒化アルミニウム焼結体または窒化アルミニウム基焼結体は公知の方法にて製造したもの用いることができる。

【0017】更に、被覆板3は、上面に凹溝11が形成されたセラミックス焼結体製の被覆下部板3aと、この凹溝11に装填された金属製の電極板4と、被覆下部板3aと電極板4の全領域を覆うセラミックス焼結体製の被覆上部板3bと、電極板4に一端が接続され、耐熱性に優れたニッケルにより形成された高周波/直流電圧印加用電極5を備えている。この電極板4には、プラズマ発生用の高周波電圧及び/又は静電吸着用の直流電圧を印加し、プラズマ発生用電極及び/又は静電吸着用電極として作用させる。

【0018】そして、被覆下部板3aと被覆上部板3bとはガラス質接合剤により気密に接合され、その接合界面には周期表第IIIa族元素から選ばれた少なくとも2種の元素と、アルミニウムと、珪素を含むオキシナイトライドガラス層が形成されており、接合後の接合層7の厚みが5~180μmとなるよう構成されている。

【0019】以下、本発明を構成する各構成要件について、詳述する。

【ガラス質接合剤】ヒータエレメント8を挟持する基体2と被覆下部板3aとの間、及び電極板4を挟持する被

覆下部板3aと被覆上部板3bとの間を気密に接合するガラス質接合層として、周期表第IIIa族元素から選ばれた少なくとも2種の元素、アルミニウム、珪素を含有するオキシナイトライドガラス層を形成させるとした理由は次のとおりであり、このガラス質接合層を形成させることにより、前記接合部7、10の気密性を大幅に向上させると共に、この気密性を長期に亘って確保し得る。

【0020】即ち、前記成分を有するオキシナイトライドガラスはセラミックス焼結体との濡れ性が良好であり、接合強度が優れ、接合部7、10の気密性が良好で、接合強度のバラツキも小さく、耐熱性にも優れている。

【0021】前記成分を含有するオキシナイトライドガラスの熱膨張係数は $3\times 10^{-6}$ ~ $8\times 10^{-6}$ /°Cであり、例えばセラミックス焼結体の中で好適に使用される窒化アルミニウム焼結体の熱膨張係数( $3.8\times 10^{-6}$ ~ $4.7\times 10^{-6}$ /°C)と一致もしくは近似し、もって繰り返しの昇温、降温の熱負荷時の熱応力による接合層7、10の破損、即ちクラックの発生を回避することができ、前記接合層7、10の気密性を長期に亘って確保することができる。

【0022】また、前記の成分を含有するオキシナイトライドガラス層のガラス軟化点Tgは850~950°Cと高く、高温雰囲気に長時間晒されても接合層7、10が劣化しない。

【0023】前記オキシナイトライドガラス層を形成し得る接合剤は、例えば以下のようにして製造することができる。まず、接合材原料粉末として、例えば周期表第IIIa族元素から選ばれた少なくとも2種の元素の酸化物と、二酸化珪素と、酸化アルミニウムとを混合するか、または熱処理によりこれらに変化する化合物を混合する。ここに、周期表第IIIa族元素の酸化物としては特に限定されずY<sub>2</sub>O<sub>3</sub>、Dy<sub>2</sub>O<sub>3</sub>、Er<sub>2</sub>O<sub>3</sub>、Gd<sub>2</sub>O<sub>3</sub>、La<sub>2</sub>O<sub>3</sub>、Yb<sub>2</sub>O<sub>3</sub>、Sm<sub>2</sub>O<sub>3</sub>等を例示することができる。

【0024】これらのうち、価格、入手のしやすさの点から、用いる周期表第IIIa族元素の酸化物の1つはY<sub>2</sub>O<sub>3</sub>が最適であり、他の周期表第IIIa族元素の酸化物はこのY<sub>2</sub>O<sub>3</sub>と全率固溶体を形成しやすいDy<sub>2</sub>O<sub>3</sub>、Er<sub>2</sub>O<sub>3</sub>、Yb<sub>2</sub>O<sub>3</sub>が好適であり、特にDy<sub>2</sub>O<sub>3</sub>は価格の点からも好適である。

【0025】また、前記各成分の組成比率も特に限定されないが、周期表第IIIa族元素から選ばれた少なくとも2種の元素の酸化物を合量で20~50重量%、二酸化珪素を30~70重量%、酸化アルミニウムを10~30重量%含む溶融体が形成されるように配合するが、得られる溶融体の融点が低く、またセラミックス部材等との濡れ性にも優れるので好ましい。更に、周期表第IIIa族元素が2種の場合は、周期表第IIIa族元素

の酸化物がモル比で約1:1となるように配合されるのが、接合材の融点が最も低くなるので好適である。

【0026】そして、上述の原料混合粉末を、例えば粒径5μm以下に粉碎し、1500~1700°Cで溶融した後急冷してガラス質の冷却物を得、これを粒径5μm程度に粉碎して均一組成の溶融体微粉末の接合剤を調整する。この接合材の調製時の雰囲気は特に限定されないが、窒素雰囲気下で行うとオキシナイトライドガラスが形成され、非窒素雰囲気下で行うとオキサイドガラスが形成される。

【0027】しかし、本発明にあっては接合界面にオキシナイトライドガラス層が形成されてなることが必須であるため、接合材の調製は窒素含有雰囲気下で行い、接合剤を予めオキシナイトライドガラス化させておくのが好ましい。窒素含有雰囲気は、N<sub>2</sub>ガス、H<sub>2</sub>、N<sub>2</sub>混合ガス又はNH<sub>3</sub>ガス等を用いることにより得られる。

【0028】また、前記接合材には、Si、N<sub>2</sub>粉末及び/又はAlN粉末を外割りで1~50重量%配合することが好ましい。即ち、Si、N<sub>2</sub>粉末やAlN粉末の添加は、このオキシナイトライドガラスの熱膨張係数を下げると共に耐熱性も向上する。配合率1重量%未満では添加する意味がなく、50重量%超では接合強度の低下をもたらすので好ましくない。添加するSi、N<sub>2</sub>粉末及び/又はAlN粉末の粒径は特に限定されないが、均一な濃度のオキシナイトライドガラスを形成させることができると点で平均粒径0.8μm以下のものが好ましい。

【0029】【接合層の厚み】基体2と被覆板3との接合後の接合層10の厚みを5~180μm、被覆下部板3aと被覆上部板3bとの接合後の接合層7の厚みを5~180μmとすることが好適な理由は、次のとおりであり、前記接合部の気密性を更に強固に確保することができる。

【0030】即ち、接合層10の厚みが5μm未満では、接合層10の端部におけるフィレットの形成が不十分であることから、接合部10の気密性を確保できず、また接合強度も不足する。一方、接合層10の厚みが180μm超では、接合層10の気密性は確保できるものの、接合強度の低下が起りやすく、また、接合時の加熱処理により溶融したガラス質接合剤が接合層10の端部から流出して基体2と被覆下部板3aとを平行に接合することができず、もって製品歩留まりが低下し、また接合作業にも支障を来たす虞がある。

【0031】また、接合層7の厚みが5μm未満では、接合層7の端部におけるフィレットの形成が不十分であることから、接合部7の気密性を確保できず、また接合強度も不足する。一方、接合層7の厚みが180μm超では、接合層7の気密性は確保できるものの、接合強度の低下が起りやすく、また、接合時の加熱処理により溶融したガラス質接合剤が接合層7の端部から流出して被覆下部板3aと被覆上部板3aとを平行に接合すること

ができず、もって製品歩留まりが低下し、また接合作業にも支障を来たす虞がある。

【0032】【ヒータエレメント】前記ヒータエレメント8の材質は、下記の理由から、添加剤が添加されることなく焼結され、焼結密度が2.8g/cm<sup>3</sup>、室温での電気比抵抗が0.1Ωcm以下の炭化珪素焼結体からなることが好ましい。そして、このヒータエレメント8に通電することにより、被覆板3を所定の温度に保持することができる。

10 【0033】このような炭化珪素焼結体からなるヒータエレメント8は、例えば、特開平4-65361号公報に記載されている概略下記のいずれかの方法で製造することができる。

① 平均粒子径が0.1~10μmの第1の炭化珪素粉末と、非酸化性雰囲気のプラズマ中にシラン化合物またはハロゲン化珪素と炭化水素とからなる原料ガスを導入し、反応系の圧力を1気圧未満から0.1torrの範囲で制御しつつ気相反応させることによって合成された平均粒子径が0.1μm以下の第2の炭化珪素粉末とを混合

20 し、これを加熱し焼結することによって炭化珪素焼結体を得て、この焼結体を所望のパターンに従って放電加工してヒータエレメントとする。

【0034】② 非酸化性雰囲気のプラズマ中にシラン化合物またはハロゲン化珪素と炭化水素とからなる原料ガスを導入し、反応系の圧力を1気圧未満から0.1torrの範囲で制御しつつ気相反応させることによって合成された平均粒子径が0.1μm以下の炭化珪素粉末を加熱し、焼結することによって炭化珪素焼結体を得て、この焼結体を所望のパターンに従って放電加工してヒータエレメントとする。

30 【0035】しかして、これらのヒータエレメント8は添加剤無添加、即ち異種物質を添加することなく焼結された炭化珪素焼結体により形成されたものであるから、均質であり、その結果局所的な異常発熱はなく、接合層7、10の一部が溶融して前記接合部にリークが生じることはなく、接合層7、10の気密性を更に強固に確保し得るものである。

【0036】また、添加剤が無添加であることから極めて高純度であり、かつ、焼結密度が2.8g/cm<sup>3</sup>以上という高密度な焼結体であることから、前記接合部にリークが生じて気密性が損なわれても、ヒータエレメント8からの添加剤、即ち不純物の蒸発はなく、反応チャンバー内が汚染される虞はない。更に、高温高強度にも優れることから熱衝撃によるヒータエレメント8の変形や断線がなく、更に室温での電気比抵抗が1Ω·cm以下という低電気比抵抗であることからヒータエレメント8を細線化、薄膜化する必要はないことから、ヒータエレメント8が断線する虞はない。

50 【0037】【電極板】電極板4に印加用電極5を介してプラズマ発生用電源から高周波電圧を印加することに

より、プラズマを発生させることが可能となる。このとき、電極板4は0.025mm以上の充分な厚みT<sub>1</sub>を持った金属板であるため、①高周波電圧を印加しても発熱して焼き切れる虞がない他、②格子状またはメッシュ状の電極を用いた場合と異なり、全域に緻密、安定、且つ均一なプラズマを発生させることができ、③更に、電極板4と印加用電極5との連結を面／ロッド間で確実に行える等の利点を有する。

【0038】また、前記電極板4に印加用電極5を介して静電吸着用電源より500V程度の直流高電圧を印加すると、被覆板3が絶縁体として機能し、シリコンウエハ等の被吸着物を静電吸着させることができるとなる。なお、電極板4にプラズマ発生用電源の高周波電圧と静電吸着用電源の直流高電圧の両方を印加する場合には、高周波をカットできるフィルタを静電吸着用電源と給電用電極間に設置すればよい。

【0039】更に、前記電極板4は、①セラミックスの熱膨張係数に近似する熱膨張係数を有すること、②接合工程中の熱処理温度及び雰囲気に安定であること、③固有抵抗値が低いこと、④室温～1000°Cまでの実用温度域での長期使用が可能である等の理由から、モリブデン、タンクステン、タンタル、ニオブ若しくはこれらの合金等の高融点金属からなることが好ましい。

【0040】〔セラミックス焼結体〕前記基体2、前記被覆下部板3a、前記被覆上部板3bの材質は特に限定されるものではないが、熱伝導性、機械的強度、耐プラズマ性に優れ、CF<sub>4</sub>、C<sub>2</sub>F<sub>6</sub>、C<sub>4</sub>F<sub>8</sub>等のプラズマクリーニングガスに対する耐久性に優れる等の理由から、窒化アルミニウム焼結体、または窒化アルミニウム基焼結体により形成されてなることが好ましい。この窒化アルミニウム焼結体や窒化アルミニウム基焼結体は公知の方法にて製造したものを用いることができる。

【0041】そして、この実施の形態に係るセラミックスヒータは、例えば、次のようにして製造することができる。表面に凹溝12を有する基体2、被覆下部板3aは、表面に凸部が形成された金型をセラミックス成形体板にプレス押圧し、焼結して形成する。焼結温度等の焼結条件は従来法に従えばよい。被覆上部板3も従来法に従って形成する。そして、電極板4を前記被覆下部板3aの凹溝11に、前記方法に従って形成されたヒータエレメント8を基体2の凹溝12にそれぞれ装填する。

【0042】次いで、微粉碎された前記ガラス質接合剤をスクリーンオイルと混合してペースト化し、このペースト状ガラス質接合剤を、凹溝12にヒータエレメント8が装填された基体2と被覆下部板3aのそれぞれの接合面、および凹溝11に電極板4が装填された被覆下部板3aと被覆上部板3bのそれぞれの接合面に塗布し、100～200°Cで乾燥する。

【0043】その後、ガラス質接合剤が塗布された面を介して、基体2と、被覆下部板3aと、被覆上部板3b

とを、ヒータエレメント8と電極板4とをそれぞれ挟持した状態で積層し、電気炉中で加熱してガラス質接合剤を溶融し、1300～1500°Cで5～40分加熱して接合する。

【0044】この加熱は常圧又は10気圧程度以下の加圧下において行なわれる。加熱時の雰囲気は用いる接合剤により異なる。即ち、オキシナイトライドガラスを含有する接合剤を用いる場合は、接合剤が予めオキシナイトライド化しているため、非窒素含有雰囲気であっても良いが、好適には窒素含有雰囲気である。これに対して、オキサイドガラスを含有する接合剤を用いる場合は、オキサイドガラスをオキシナイトライド化するための窒素源が必要となり、窒素含有雰囲気で行う。なお、窒素含有雰囲気は、N<sub>2</sub>ガス、H<sub>2</sub>～N<sub>2</sub>混合ガスまたはN<sub>2</sub>H<sub>4</sub>ガス等を用いることにより得られる。

【0045】加熱溶融した接合剤は冷却固化することにより接合強度を持ち得るようになるが、急速冷却を行わず徐冷することにより、窒素の取り込み量を高い状態で保持しつつオキシナイトライドガラス層の安定化を図ることが好ましい。冷却速度は50°C/min以下が好ましく、更に好ましくは30°C/min以下である。

【0046】なお、接合後の接合層7、10の厚みをそれぞれ前記の範囲とするためには、微粉碎されたガラス質接合剤とスクリーンオイルとの量比、ペースト状ガラス質接合剤の塗布量、接合時の加熱温度、時間等の処理条件を適宜調整することにより行うことができる。

【0047】

【実施例】以下、実施例を詳述する。

【実施例1～5】表面に幅5mm、深さ3mmのヒータエレメント装填用のスパイラル状凹溝が設けられた直径220mm、厚み15mmの窒化アルミニウム焼結体製の基体と、表面に直径200mm、深さ0.3mmの電極板装填用の円盤状凹溝が設けられた直径220mm、厚み8mmの窒化アルミニウム焼結体製の被覆下部板と、直径220mm、厚み1mmの窒化アルミニウム焼結体製の被覆上部板を従来法に従って形成した。

【0048】一方、焼結助剤や導電性を付与するための添加剤が添加されることなく焼結され、焼結密度が3.1g/cm<sup>3</sup>、室温での電気比抵抗が0.05Ω·cmの炭化珪素焼結体からなり、前記スパイラル状凹溝に装填し得る形状のヒータエレメントを、前記段落番号【0031】の製造方法①により形成した。第1の炭化珪素粉末の平均粒径は0.7μm、添加量は95重量%、第2の炭化珪素の平均粒径は0.01μm、添加量は5重量%であり、ホットプレス焼結条件はプレス圧400kg/cm<sup>2</sup>、焼結温度2200°C、焼結時間90分である。また、電極板として、直径200mm、厚み0.3mmのモリブデン金属板を用意した。

【0049】次いで、表1に示す組成を有するガラス質接合剤(粒径約2μm)を市販のスクリーンオイルと混

合してペースト化し、このペースト状ガラス質接合剤を、前記スパイラル状凹溝に前記ヒータエレメントが装填された前記セラミックス焼結体製基体と前記セラミックス焼結体製被覆下部板のそれぞれの接合面、及び前記円盤状凹溝に前記モリブデン製電極板が装填された前記セラミックス焼結体製被覆下部板と前記セラミックス焼結体製被覆上部板のそれぞれの接合面に塗布し、100～200°Cで乾燥した。

【0050】その後、ガラス質接合剤が塗布された面を介して、前記セラミックス焼結体製基体下部板と、前記セラミックス焼結体製基体上部板と、前記セラミックス焼結体製被覆板と、前記ヒータエレメントと前記電極板とをそれぞれ挟持した状態で積層し、N<sub>2</sub>ガス雰囲気の電気炉中で加熱してガラス質接合剤を溶融し、1400°Cで20分加熱して気密に接合した。冷却速度は25°C/min、接合後の接合層の厚みは共に50μmであった。また、接合界面にはオキシナイトライドガラスが形成されていることを、オージェ電子分光法により確認し\*

	接合剤原料粉末組成(重量%)				気密性
	Dy <sub>2</sub> O <sub>3</sub>	Y <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	
実施例1	15	10	25	50	○
実施例2	15	15	25	45	○
実施例3	15	15	20	50	○
実施例4	20	20	20	40	○
実施例5	25	25	15	85	○

【0054】【比較例1～4】実施例1～5に準じてセラミックスヒータを作製し、耐久性試験に供した。ただし、ガラス質接合剤の組成は表2に示すとおりである。※

	接合剤原料粉末組成(重量%)				気密性
	Dy <sub>2</sub> O <sub>3</sub>	Y <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	
比較例1	0	20	40	40	×
比較例2	0	35	20	45	△
比較例3	0	40	10	50	×
比較例4	20	0	40	40	×
比較例5	35	0	20	45	△
比較例6	40	0	10	50	×

#### 【0056】

【発明の効果】以上のように、本発明のセラミックスヒータにあっては、セラミックス焼結体製の基体と、この基体の接合面の全領域を覆うセラミックス焼結体製の被覆板とを備えるとともに、前記基体と前記被覆板との間に把持されたヒータエレメントと、このヒータエレメントの一端が接続された少なくとも1対のヒータ給電用電極とを備え、前記基体と前記被覆板とは接合剤により気密に接合され、その接合界面には周期表第IIIa属元素から選ばれた少なくとも2種の元素と、アルミニウムと、珪素とを含むオキシナイトライドガラス層が形成されていることにより、接合部の気密性を大幅に向上で

き、その気密性を長期に亘って確保できて、反応チャーバー内の汚染を防止し、また接合部の気密性が損なわれても反応チャーバー内の汚染を生じることがなく、ヒータエレメントの破断を防ぎ、製品寿命を長くすることができる。

【0057】また、請求項2に係るセラミックスヒータでは、前記ヒータエレメントは添加剤が添加されることなく焼結され、焼結密度が2.8g/cm<sup>3</sup>以上、室温での電気比抵抗が0.1Ωcm以下の炭化珪素焼結体からなることにより、接合部の気密性が損なわれた場合にあっても、添加剤の蒸発等により引き起こされる反応チャーバー内の汚染を生じることがなく、さらにまた高温強度

